Performance Monitoring Protocol (QA/QC) for the Agilent Headspace GC/MS

1 Scope

This document addresses the performance monitoring (QA/QC) of the Agilent Gas Chromatograph/ Mass Spectrometer (GC/MS) system with a Headspace Autosampler, which may include optional detectors. This document applies to personnel using the associated instrument(s)/equipment in the following discipline/category of testing: Explosives chemistry examinations performed at the Huntsville facility.

2 Principle

The Agilent Headspace GC/MS is a gas chromatograph (GC) with a headspace autosampler and a single quadrupole Mass Selective Detector (MSD) Mass Spectrometer. The system may also be equipped with an additional detector. A headspace autosampler is a device used to sample the gas phase volatile analytes within a sealed vial. This sampling is transferred to the inlet of the GC and onto a column where the components are separated and sent to the detector. There may be two columns in the Agilent GC (labeled front and back), each leading to respective detectors. The front column is a capillary column which leads to a single quadrupole Mass Selective Detector (MSD) Mass Spectrometer. The MSD is configured with a dedicated electron impact ionization (EI) source in the mass spectrometer, and is referred to as the HS-MSD throughout this document.

This performance monitoring protocol is based upon the manufacturer's recommendations. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

3 Equipment/Materials/Reagents

a. Instrumentation - Agilent 7890B Gas Chromatograph, Agilent 5977A Mass Selective Detector with EI Source, Chemstation Software, and/or Gerstel Maestro Software (or equivalent)

b. Autosampler - Gerstel "MPS2" ""automated sampler, accessories, and software (or equivalent)

c. Capillary GC Columns: Agilent DB-624, 30 m, 0.25 mm i.d., 1.4 μm film (or equivalent)

d. Carrier Gases: Helium, 99.99% (high purity or equivalent)

e. Perfluorotributylamine (PFTBA, FC-43) (Agilent or equivalent)

Redacted

m. Deionized Water, 18.2 MΩ Milli-Q (or equivalent)

n. Autosampler vials - 10 or 20 mL crimp-top headspace autosampler vials or appropriate headspace vials for “”Gerstel Series autosamplers (Gerstel or equivalent)

o. Injection port septa - standard low-bleed 11 mm (Agilent or equivalent)

p. Injection port liners - split-splitless, tapered, with or without glass wool (Agilent or equivalent)

q. Autosampler syringes - 2.5 mL headspace and 1 mL liquid syringes (Gerstel or equivalent)

4 Standards and Controls

The Testmix is used to assess daily operating performance and continued integrity of the system. It will be analyzed and evaluated prior to the analysis of evidence.

4.1 Testmix

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Refer to the analyte-specific SOP for unique samples, Redacted for the preparation of the positive control standard which will be used as the Testmix. Record stock solution preparations in the Reagent Log.

4.2 PFTBA Tuning Solution

The PFTBA tuning solution is used for tuning the mass spectrometer and verifying mass calibration. It is supplied by the instrument manufacturer and does not expire. It is stored in a glass container attached to the MSD. Refill as needed.

5 Sampling or Sample Selection

Not applicable.

6 Procedures

6.1 Daily Checks – HS-MSD

The following steps are to be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

a. Record the remaining disk space on the hard drive. Use Windows to verify that the hard disk has at least 100 MB of free disk space. Do not use if less than 100 MB remain.

b. Record the line pressure of the building helium supply (carrier gas). The regulator should read 50 psi or above. If it cannot maintain this pressure, contact the appropriate instrument support personnel. If the helium is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100 psi. remain.

c. Perform a tune of the instrument. If Autotune (ATUNE) is selected, the mass spectrometer will tune itself using PFTBA. Evaluate the results using the 'Decision Criteria' section of this protocol. Ensure N_2 and H_2O values do not exceed 5%. If the results are acceptable print the tune file (ATUNE) when completed.

d. Check the Ion Gauge to ensure that there are no significant leaks in the system. Do not use if the pressure is higher than 6×10^{-5} torr.

e. Refer to the analyte-specific SOP for unique samples, Redacted for the appropriate procedure, instrumental conditions, and decision criteria for performing an analysis of the Testmix. For volatiles analysis, perform an analysis of the headspace above the Testmix prior to the analysis of evidence. Open the appropriate

Testmix instrument method, and verify the parameters as listed in the 'Instrumental Conditions' section of this protocol. Set up a sequence, load the autosampler with a vial containing the appropriate Testmix, and start the analysis. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the TIC and a representative mass spectrum.

f. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact the appropriate instrument support personnel.

6.2 As Needed Checks

The following steps are to be performed as needed based on system performance. Indicate completion in the appropriate log.

a. Replace the septum in the GC injection port.

b. Replace the liner within the GC injection port.

c. Check the syringe in the autosampler. Replace if needed.

d. Check the internal bungee cords in the autosampler. Replace if needed.

e. Check the plungers in each autosampler syringe. Replace if needed.

7 Instrumental Conditions

7.1 HS-MSD Testmix Parameters

7.1.1 Headspace Sampler Parameters

Incubation temperature:	80°C
Incubation time:	5 min
Agitator speed:	300 RPM
Agitation timing:	10 sec on 1 sec off
Syringe temperature:	90ºC
Sample fill volume:	1.0 mL
Sample fill rate:	1.0 mL/sec
Sample fill strokes:	5
Sample injection speed:	1.0 mL/sec
Syringe flush time:	4.0 min

7.1.2 Gas Chromatograph Parameters

Oven

Temperature:	40ºC for 4 min
Ramp:	10ºC/min to 120ºC for 0 min
Run time:	12 min
Equilibration time:	0.25 min

Column

Type:	DB-624
Length:	30 m
Internal diameter:	0.25 mm
Film thickness:	1.4 µm

Inlet/Carrier

Inlet temperature:	150ºC
Injection mode:	Split
Carrier gas:	Helium, 99.99% (split)
Carrier mode:	Constant pressure
Pressure:	5.3 psi
Split ratio:	10:1

7.1.3 Mass Spectrometer Parameters

Ionization mode:	Electron impact
Scan mode:	Full scan
Scan range:	29 – 400 amu
Relative voltage:	106 V
Source temperature:	230ºC
Transfer line temperature:	260ºC
Quadrupole temperature:	150ºC
Solvent delay:	2.0 min

8 Decision Criteria

8.1 HS-MSD Testmix

Verify the results of the Testmix.

a. In order for the instrument to be considered in good operating condition, all Testmix components should generate well-resolved, Gaussian-shaped peaks with baseline separation.

b. A SNR of 3:1 will be the minimum response necessary to consider a response a peak.

c. There should be no extraneous peaks in the chromatogram/TIC greater than 5% of the height of the tallest Testmix peak.

d. The retention times of the Testmix components should not deviate by ±3% compared to previous runs of the Testmix.

e. Check for the correct mass assignments for the mass spectra. In order for the MS to be considered in good operating condition, the correct mass assignments for each of the analytes in the appropriate Testmix should be present. Redacted

f. Compare the mass to charge ratios to previous Testmix spectra and library spectra.

8.2 MSD Tune

Verify the results of the tune. Compare the results of the tune to previous tune results. Significant voltage increases or changes in the isotope ratios indicate the need to initiate corrective maintenance procedures. The following are typical ATUNE values for the MSD:

a.	PFTBA Tune:	Mass ±0.4 for m/z 69, 219, and 502
b.	Peak width:	0.45-0.65
c.	Relative abundance:	69 greater than 50% 219 greater than 50% 502 greater than 1%

9 Calculations

Not applicable.

10 Measurement Uncertainty

Not applicable.

11 Limitations

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

12 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis. Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

13 References

Manufacturer(s)'s Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (IOG 001) *Instrument Operations Group SOP Manual.*

"Gas Chromatograph General Maintenance Protocol" (IOG 002) *Instrument Operations Group SOP Manual.*

"Mass Spectrometer General Maintenance Protocol" (IOG 004) *Instrument Operations Group SOP Manual.*

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
0	10/04/18	New document which specifies instrument protocol for the Huntsville facility.

Redacted - Signatures on File

Approval

Scientific Analysis
Unit Chief Date: 10/03/2018

TL Approval

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QA Approval

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